STUDY TITLE

Acute Toxicity Evaluation of Step I Pipe Discharge (WS006/WS007) from US Steel-MinnTac, Mountain Iron, Minnesota

DATA STANDARD

"Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms," 2002, Fifth Edition. EPA/821/R/02/012. U.S. Environmental Protection Agency, Washington, DC.

STUDY DATES

November 21-23, 2016

STUDY SUBMITTED

December, 2016

TESTING FACILITY

Pace Analytical 4730 Oneota Street Duluth, MN 55807

Tel. No. (218) 727-6380

PROJECT IDENTIFICATION NUMBERS

Pace Project # 1279287

NPDES Permit No. MN0057207

1.0 EXECUTIVE SUMMARY AND RESULTS

MinnTac Step I Pipe Discharge (WS006/WS007), Mountain Iron, MN was evaluated for acute toxicity to fathead minnows (*Pimephales promelas*) during November 21-23, 2016. McNiven Creek Water (SW002) was used as the dilution and primary control water for the test. Moderately hard reconstituted water (MHRW) was used as the laboratory reference control water for the tests. The table below summarizes the toxicity results of the tests.

Fathead Minnow

48 Hour LC₅₀: >100% (95% C.I. Not Calculable) Survival NOEC: 100% Survival LOEC: --Toxicity Units (TUa): <1.00

The chemistry determinations for the final effluent samples are attached at the end of this report. The data package and statistical analysis for the test battery is also attached.

2.0 GENERAL INFORMATION

Data Standard	EPA/821/R/02/012								
Testing Facility	Pace Analytical, 4730 Oneota Street, Duluth, MN 55807, Tel. No. (218) 727-6380								
Project Director	Dan Toms	Dan Toms							
Test Dates	November 21-23, 2016								
Report Approval	Dan Toms	Report Signature:							

3.0 OBJECTIVE

To determine the median lethal concentration (LC₅₀), no- & lowest- observed effect concentrations (NOEC & LOEC) based on survival of MinnTac Step I Pipe Discharge (WS006/WS007) for fathead minnows.

4.0 REFERENCES

American Public Health Association (APHA). 1998. 20th Ed. of Standard Methods for the Examination of Water and Wastewater. APHA, Washington, D.C.

USEPA. 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. EPA/821/R/02/012. U.S. Environmental Protection Agency, Washington, DC.

MPCA Acute Toxicity Test Conditions for Static Renewal Whole Effluent Tests. Minnesota Pollution Control Agency.

West Inc. & University of Wyoming. TOXSTAT Software Package Version 3.5.

5.0 TEST METHODS

Test Concentrations: 0 (dilution water control), and 100%.

Test Organisms: Fathead minnow (Pimephales promelas) 6 days old.

Test Chambers: Fathead minnow: 800 mL glass beakers.

Incubation: Duration: 48 hours. Renewals: None

Observations: Survival was recorded each day. The test was terminated following 48 hours of exposure.

Endpoint Calculations: LC₅₀ USEPA Statistical Programs. NOEC and LOEC: TOXSTAT statistical software,

Version 3.5, West, Inc., and University of Wyoming.

6.0 DEVIATIONS FROM REFERENCED METHOD

To the best of our knowledge, no deviations from the referenced method (EPA-821-R-02-012) occurred for the study.

Cumulative Percentage of Surviving Organisms for Selected Test Species Exposed to MinnTac Step I Pipe Discharge (WS006/WS007) Tested November 21-23, 2016

Effluent Concentration	Temperatura de la companya del companya de la companya de la companya del companya de la companya del companya de la companya de la companya de la companya del companya de la companya del companya de la companya de la companya del companya de la companya del companya del companya del companya del companya	Test Interval
(%)	24 hour	48 hour
	Fathead Minnow	
0 (MHRW) ^a	100° (20/20) ^d	100° (20/20) ^d
0 (RW) ^b	100° (20/20) ^d	100° (20/20) ^d
100	100° (20/20) ^d	100° (20/20) ^d

^a Secondary Control was Moderately Hard Reconstituted Water (MHRW)

^b Primary Control Water was McNiven Creek Water (SW002)

^c Cumulative Percentage Survival

d # Alive/# Exposed



Acute Whole Effluent Toxicity Test Report

NPDES Permit Program

Doc Type: Effluent Limit Standards Review

Please read all instructions carefully before completing each section of the report. Instructions are found on Page 5.

Address the completed, signed report to:

Attn: Water Quality Submittals Minnesota Pollution Control Agency 520 Lafayette Road North St. Paul, Minnesota 55155-4194

Facility Information Permit No.: MN0057207 Station ID: WS006/WS007 Name of permittee: US Steel Minntac Test Information Test end date (mo/day/year): 11/23/16 Test start date (mo/day/year): 11/21/16 or ☐ Check if no discharge occurred and leave the rest of the form blank. Report completion date (mo/day/year): 12/1/16 WET test type (check one): ☐ Initial ☐ Repeat #1 ☐ Repeat #2 ☐ TAC Repeat For repeats, test start date of initial WET test (mo/day/year): Dilution water (check one): Lab Receiving water If receiving water, indicate name: McNiven Creek Water (SW002) Phone: (218) 727-6380 Test lab name:: Pace Analytical Mailing address: 4730 Oneota Street 55807 City: Duluth State: Minnesota Zip code: E-mail: dan.toms@pacelabs.com Lab representative name: Dan Toms E-mail: tmoe@uss.com Facility representative name: Thomas Moe

Sample condition upon test initiation

linity Hardness 3/L) (mg/L)	20.7 28.0				259 1100							
Total Ammonia Alkalinity (mg/L) (mg/L)	< 0.20 20		:		1.2 25							
Temp °C upon test initiation (25				25							
Temp °C Arrival at lab	0.4				4.				r Liter (mg/L.)			of grabs: of grabs: of grabs:
TRC (mg/L)	< 0.02			-	< 0.02				mg/L = milligrams per Liter (mg/L)			- Enter number - Enter number - Enter number - Enter number
Conductivity (umhos/cm)	65.5				2130					e mesh size:		Grab-composite – Enter number of grabs:
PH (SU)	7.0				8.2	i			umhos/cm = micromhos per centimeter eck one)	stal		
First use date	11/21/16				11/21/16				s/cm = microm	k each one the Effluent 3 □ □ Yes 図 No	each sample):	Grab Grab
Receipt date	11/21/16				11/21/16				*If applicable umhos/cm = micromhos per Does facility have chlorine limit? (check one)	Was sample dechlorinated at lab? (check each one that applies) ☐ Effluent 1 ☐ Effluent 2 ☐ Effluent 3 ☐ Effluent 4 Effluent filtered? (check one) ☐ Yes ☒ No If yes,	Effluent sample type (check one type for each sample):	☐ 24 hr composite☐ 25
Collection	11/21/16				11/21/16				*If applicable cility have chlorine	le dechlorinatec iffluent 1 E ent_filtered? <i>(c</i>)	nple type (<i>chec</i>	Effluent 1: 2 Effluent 2: 2 Effluent 3: 2 Effluent 4: 2
Sample	Receiving Water 1	Receiving Water 2*	Receiving Water 3*	Receiving Water 4*	Effluent 1	Effluent 2*	Effluent 3*	Effluent 4*	*If &	Was sampl □ E	Effluent sar	Efflu Efflu Efflu

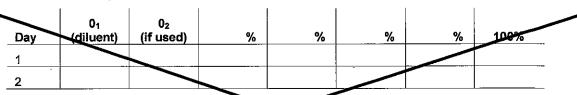
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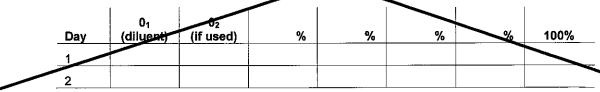
Summary of data for Fathead Minnows - Percent survival per concentration

Day	0 ₁ (diluent)	0 ₂ (if used)	%	%	%	%	100%
1	100	100					100
2	100	100					100
3							
4							





Summary of data for Daphnia magna - Percent survival per concentration



Summary of results - Acute values

Species	LC ₅₀	TU _a (acute toxic units)
Fathead Minnow (96 hour)	>100%	<1.00
Ceriodaphnia dubia (48 hour)		
Daphnia magna (48 hour)		

Test Acceptability Criteria (TAC)

QA/QC Criteria	Criteria met for Acute Toxicity
Fathead Minnow – Age range <24 hours at test start	☐ Yes No
Ceriodaphnia dubia - Age range <24 hours at test start	☐ Yes ☐ No
Daphnia magna – Age range <24 hours at test start	☐ Yes ☐ No
Test started within 36 hours of collection of sample	⊠ Yes □ No
90% or greater survival of all control organisms (if any species results fail, check No)	⊠ Yes □ No

If reference toxicant other than NaCI used, please indicate:

Test deviation comments

Describe any deviation from test methods:

Per permit requirements only 48 hour fathead minnow test performed with no dilution.

(For example: pH-controlled test amount of CO₂, reduced DO levels in test leading to aeration, sample exceeded holding time.)

Certifications

Form shall be signed by the laboratory here	certifying the results:	
Lab representative (print): Dan Toms	Phone:	(218) 727-6380
Signature: an //www	Date:	12/1/16
Form shall be signed by the permittee here i Applications and Reports	n accordance with Minn. R. 7001.0	540 Certification of Permit
I certify under penalty of law that this document in accordance with a system designed to assure submitted. Based on my inquiry of the person of responsible for gathering the information, the in accurate, and complete. I am aware that there a possibility of fine and imprisonment.	e that qualified personnel properly ga r persons who manage the system of formation submitted is, to the best of	ther and evaluate the information r those persons directly imy knowledge and belief, true,
Permittee representative (print): Tom Moe	Phone:	(218) 749-7485
Signature:	Date:	
Attachments provided:		
Data package. Narrative report. Reference Toxicant	Chart (Reference test met requirements)	

Whole Effluent Toxicity Test Instruction Sheet

This instruction sheet is provided to aid in filling out the Acute or Chronic Whole Effluent Toxicity (WET) Test report forms as required by your National Pollutant Discharge Elimination System (NPDES) Permit. If your permit requires acute WET testing, there will be one form to fill out. If your permit requires chronic WET testing, there will be two forms to fill out.

Section 1. Facility Information

Fill out your NPDES permit number, discharge outfall station identification (ID) number, and your Permittee name as it appears on your NPDES permit.

Section 2. Test Information

Test Start Date/Test End Date. Fill out the date the test began (month/day/year), the date the test finished (month/day/year)

No discharge. This box should be checked if no discharge occurred during the WET monitoring period. If no discharge occurred, the rest of the form is left blank, and should be signed, dated, and submitted by the required date.

Report Completion Date. Fill out the completion date, which is the date the report is sent from the testing laboratory as indicated on the test report (month/day/year).

WET test type. Select the appropriate form to fill out. This will be either the acute test form, or both the Fathead chronic minnow form and the *Ceriodaphnia* chronic form. Check the appropriate test represented by this report; either Initial, Repeat#1, Repeat#2, or TAC Repeat

Dilution water. Check either lab or receiving water, whichever was used to dilute the effluent sample to create the test series concentrations. If receiving water is used as the diluents, supply the name of that receiving water.

Test Lab Name, etc. Provide the name, phone number, address, lab representative name, and e-mail address.

Facility representative name. Provide the name of the discharging facility's representative and e-mail address.

Sample condition table. The laboratory conducting the tests must fill in the sample information and dates associated with the samples received. The information is typically collected for Chain of Custody forms and for initial sampling upon initiating a test. Provide the date the sample was collected, receipt date at the lab, date of first use for testing, pH, specific conductivity, total residual chlorine (TRC) analysis, temperature upon arrival at the lab and temperature at test initiation, total ammonia analysis, total alkalinity, and total hardness. Sample data will be provided for each sample received and as specified by the applicable acute or chronic manuals, or by the permit. Up to four samples each may be received for the receiving water and effluent for acute tests. Up to three samples may be received for chronic tests for the receiving water. Fill in any data that is below quantification levels with a less than sign and that actual quantification level (e.g. <40 ug/l) – do not report ND.

Does facility have a chlorine (TRC) permit limit? Check either Yes or No.

Was sample dechlorinated at the lab? Check each sample that was dechlorinated at the lab.

Effluent filtered? Check either Yes or No, and state the mesh size used for filtration.

Effluent sample type. For each sample collected check or check whether the sample was a twenty-four hour composite, a grab, or a grab composite with the number of grabs taken to comprise the composite sample.

Summary of data for each species for acute or chronic tests, as applicable in your permit. For each species in an acute test summarize the cumulative survival data over all replicates for each treatment level for each day of the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (0₁) based on your answer in section 2 Test Information – Dilution water.

For a Fathead minnow chronic test summarize the cumulative 96 hour survival data over all replicates, the cumulative mean 7-day biomass data, and the 7-day cumulative mean percent survival data for each treatment level for the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (0₁) based on your answer in section 2 *Test Information — Dilution water*.

For a *Ceriodaphnia* chronic test summarize the cumulative 48 hour survival data over all replicates, the cumulative mean 7-day reproduction/female data, and the 7-day cumulative mean percent survival data for each treatment level for the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (0₁) based on your answer in section 2 *Test Information — Dilution water*.

Summary of results table for each species for acute or chronic tests, as applicable. For acute tests provide the appropriate 48 or 96 hour LC50 values for each species and the calculated toxic unit values (TUa for acute or TUc for chronic tests).

For Fathead minnow chronic tests provide the calculated IC₂₅ for larval survival and growth at the end of the test as well as the critical No Observed Effect Concentration (NOEC) and Lowest Observed Effect Concentration (LOEC). Also provide the chronic toxic units (TUc) calculated as the inverse of the IC₂₅ value.

For Ceriodaphnia chronic tests provide the calculated IC_{25} for survival and reproduction at the end of the test as well as the No Observed Effect Concentration (NOEC) and Lowest Observed Effect Concentration (LOEC). Also provide the chronic toxic units (TUc) calculated as the inverse of the IC_{25} value.

Test acceptability criteria. For acute tests check the corresponding boxes either yes or no for each of the Quality Assurance/ Quality Control (QA/QC) criteria.

For chronic tests check the corresponding boxes either yes or no for each of the QA/QC criteria. If there is deviation from the specified methods, please describe the change and reason for the deviation. PMSD is the percent minimum significant difference from hypothesis testing that characterizes overall test variability. The temperature variation of ±1° C is for individual measured test chambers over a test.

Deviations from test methods. If there is any deviation from the specified methods, please describe the change and the reason for the deviation. This is a narrative comment area to describe any unusual circumstances in conducting any of these tests.

Certifications. Provide the laboratory representative name, signature, phone number and date for the tests completed. Also provide the permittee representative name, signature, phone number and date for the tests completed. The Permittee is responsible for forwarding these results to the Minnesota Pollution Control Agency.

Attachments. Attach any narrative regarding results or SOP, bench sheets for analytical and biological data, chain of custody forms, reference test results for tested species (tables and/or charts over the past year), and toxicity calculation outputs.

www.pca.state.mn.us • 651-296-6300 • 800-657-3864 • TTY 651-282-5332 or 800-657-3864 • Available in alternative formats

CHAIN-OF-CUSTODY / Analytical Request Document the Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be

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Pace Analytical*

Document Name:

Sample Condition Upon Receipt Form

Document No.: F-DUL-C-001-Rev.01 Document Revised: 22Jan2016

Page 1 of 1

Issuing Authority:
Pace Virginia, Minnesota Quality Office

Sample Condition Client Name: Project #: **Upon Receipt** US Steel UPS USPS Courier: Fed Ex Client Other:_ Pace Commercial Tracking Number: Optional: Proj. Due Date: Proj. Name: Custody Seal on Cooler/Box Present? Yes No Seals Intact? Yes No Packing Material: Bubble Wrap Bubble Bags None Other: Temp Blank? Yes No hermometer Used: B00051
Su2007
Type of Ice: Wet Blue None
Cooler Temp Read °C: 2.0 1.0 Cooler Temp Corrected °C: 1.4 0.4 - 350 013 013 Thermometer Used: B00051 Samples on ice, cooling process has begun Comments: Chain of Custody Present? Yes □No □N/A Chain of Custody Filled Out? Yes □No □N/A 2. □No 3. Chain of Custody Relinquished? ∏N/A Sampler Name and Signature on COC? Yes □No □N/A □No Samples Arrived within Hold Time? Yes □N/A PH, res Cl Short Hold Time Analysis (<72 hr)? Tyes □No □N/A □N/A Rush Turn Around Time Requested? Yes No Sufficient Volume? **√**Yes □No □N/A 8, Yes □No □N/A 9. Correct Containers Used? -Pace Containers Used? Yes ■No □N/A □No □N/A Containers Intact? Yes 11. Note if sediment is visible in the dissolved containers. Filtered Volume Received for Dissolved Tests? Yes □No SW/A Sample Labels Match COC? Yes □No □N/A 12. -includes Date/Time/ID/Analysis Matrix: WT See pH log for results and additional preservation □N/A All containers needing acid/base preservation will be Yes ∐Nο documentation checked and documented in the pH logbook. □Yes **M**N/A Headspace in Methyl Mercury Container 13. ŪN/A Headspace in VOA Vials (>6mm)? Yes ∏No 14. Trip Blank Present? Yes □No III N/A 15. II)N/A □No Trip Blank Custody Seals Present? Yes Pace Trip Blank Lot # (if purchased): CLIENT NOTIFICATION/RESOLUTION Field Data Required? Yes No Person Contacted: Date/Time:

FECAL WAIVER ON FILE Y N

Comments/Resolution:

TEMPERATURE WAIVER ON FILE Y N

Project Manager Review: Date: 1-21-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)

Pace Analytical Precision of NaCl Acute Reference Toxicant Testing for *Pimephales promelas* (Fathead Minnows)

Date	LC ₅₀ (g/L)	Mean (g/L)	SD	CV (%)	Lower Limit (g/L)	Upper Limit (g/L)
6/28/16	6.83					
7/6/16	7.07	6.95				
7/12/16	6.82	6.91	0.14	2.0	6.62	7.19
7/19/16	6.83	6.89	0.12	1.8	6.64	7.13
7/26/16	6.60	6.83	0.17	2.4	6.50	7.16
8/2/16	6.74	6.82	0.15	2.2	6.51	7.12
8/11/16	5.95	6.69	0.36	5.3	5.98	7.40
8/16/16	7.03	6.73	0.35	5.2	6.03	7.43
8/23/16	7.07	6.77	0.35	5.1	6.08	7.46
8/30/16	6.59	6.75	0.33	4.9	6.09	7.42
9/8/16	6.37	6.72	0.34	5.0	6.05	7.39
9/13/16	7.07	6.75	0.34	5.0	6.08	7.42
9/21/16	7.07	6.77	0.33	4.9	6.11	7.44
10/4/16	7.07	6.79	0.33	4.9	6.13	7.45
10/11/16	7.07	6.81	0.33	4.8	6.16	7.46
10/19/16	6.95	6.82	0.32	4.6	6.19	7.45
10/27/16	6.99	6.83	0.31	4.5	6.21	7.45
11/1/16	7.07	6.84	0.31	4.5	6.23	7.45
11/17/16	7.03	6.85	0.30	4.4	6.25	7.45
11/21/16	6.83	6.85	0.29	4.3	6.27	7.44

MinnTac

Pace Analytical

Client:	MinnTac
Pace Pr	roject #: 1279287
Test:	Acute Toxicity Evaluation
Test Init	iation Date: November 21, 2016
Test Ter	mination Date: November 23, 2016

ENVIRONMENTAL SAMPLE TEST INFORMATION

Date: November 21, 2016
Client: MINNTAC
Pace Project #: 1279287
Dilution Water: SW002
Test Chamber: 800 mL glass
Food: None
Temperature: 25°C
Test Organism(s)/Age: Fathead Minnow/6 day
Comments:

TOXICITY TEST RENEWAL FORM

CLIENT: MINNTAC	PACE PROJECT #: 1279287
TEST: Acute Toxicity Evaluation	TEST INITIATION DATE: November 21, 2016
ORGANISM: Fathead Minnow	TERMINATION DATE: November 23, 2016

TEST DAY	0 Test Initiation	1	2	3	4
DATE	 11/21/16 	11/22/16	1/23/16		
TIME OF READING				-	
	1715	1635	IG 15	FHM	FHM
TIME OF FEEDING					
	RW	RW	RW	RW	RW
DILUTION WATER	MHRW 16-060	MHRW	MHRW	MARW	MHFW
INITIALS	CJA	CJA	CJA	/	

INITIAL CHEMISTRIES

CLIENT: MINNTAC	Pace Project #:	1279287	
TEST: Acute Toxicity Evaluation	TEST INITIATION	DATE: Nove	ember 21, 2016
ORGANISM(S): Fathead Minnow	TEST TERMINATI	ON DATE:	November 23, 2016
		167	olanian lataz ilentika karakan

	11/21/16	C28 / /	₩ A	7	
CONCENTRATION:	MHRW				
z pH (su)	8.10				
DO (mg/l)	8.6				
Cond (umhos/cm)	292				_
CONCENTRATION:	SW002	Receiving Water			
pH (su)	7.04				
DO (mg/l)	9.0	-			
Cond (umhos/cm)	73				
CONCENTRATION:	100% (V	VS006/007) 100% Ef	fluent		
pH (su)	8.24				
DO (mg/l)	8.6				
Cond (umbos/cm)	2140				

Daily Temperatures

#2

25.5

FINAL CHEMISTRIES

İ	1122/16 / 617	11123116 / CLC /
CONCENTRATION:	MURW	
pH (su)	8,06	7,91
DO (mg/l)	8.0	8.0
CONCENTRATION:	SW002 Receiving Water	
pH (su)	7.39	7.54
DO (mg/l)	7.9	7,9
CONCENTRATION:	100% (WS006/007) 100% Effluent	
pH (su)	8.31	8.37
DO (mg/l)	7.9	7.9
Daily '	Temperatures	
Bath# △		

ath#]

25,5

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017 11/30/16

ACUTE TOXICITY DATA LOG

Client: MINNTAC
Project #: 1279287
Test: Acute Toxicity Evaluation
Test Initiation Date: November 21, 2016
Investigator: Toms
Test Duration: 48 hour
Renewal: **None** / Daily / Other

Species: Fathead Minnow
Age: 6 day
No. Animals/No. Reps: 10/2
Sources of Animals: ABS
Dilution Water/Control: River Water
Test Volume: 600 mL
Test Temperature: 25°C
Minimum Control Survival ≥ 90%:(Y) N)

Concentration	Survival Readings: (# alive out of # exposed from above unless shown otherwise).						
	24 F Repl			Hour licate B	72 Hour Replicate		96 Hour Replicate A B
MHRW	10	10	01	10		/	
SW002 (RW)	10	10	10	10		\bigwedge	\
WS006/007 (100% Eff)	10	16	01	10			
	Dated Initials	CJA	Dated Initials	CJA	Dated Initials	Dated	Initials
Comments:					7		